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(54) LIQUID DETERGENT COMPOSITIONS

(71) We, UNILEVER LIMITED, a company organised under the laws of Great Britain, of Unilever House, Blackfriars, London, EC4, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to liquid detergent compositions which are suitable for fabric washing, and in particular to such compositions containing phosphate detergency builders.

Aqueous built liquid detergent compositions are well known in the art. Although they offer several advantages over other forms of detergent compositions like powders, such as improved solubility and easier dosing, their formulation is very difficult, since they should be physically stable under a wide range of storage conditions, for example from about -4°C to 37°C. Also they should have satisfactory pouring properties despite containing relatively high concentrations of detergent ingredients, especially builder salts so that ideally they have performances equivalent to the usual powdered detergent compositions.

The prior art mainly teaches the use of more water soluble ingredients, such as tetrapotassium pyrophosphate, usually with expensive stabilising agents, but even then the amount which can be used is lower than would be desired in comparison with powdered detergent compositions. Although such prior liquid formulations may provide acceptable compositions as far as their physical characteristics are concerned, their detergency and other washing properties tend to be unsatisfactory. For example, with the alkali metal pyrophosphates there are often deposits formed on the clothes, especially when the products are used at exceptionally low product concentrations, or at high temperatures. When attempts have been made to improve the detergency, then the stability and pourability have become unsatisfactory. In particular, it has

not been possible to incorporate into the liquid detergent compositions sufficiently high levels of the conventional detergency builders such as sodium tripolyphosphate without causing severe problems of separation or solidification of the liquid compositions.

It has now been found that homogeneous, clear, built liquid detergent compositions can be obtained by using certain mixtures of sodium tripolyphosphate and sodium or potassium orthophosphate as builders, and using a hydrotrope system which comprises at least two hydrotrope agents, which will be defined hereinafter.

Insofar as the two detergency builder materials function normally in different ways, i.e. by sodium tripolyphosphate acting as a sequestrant detergency builder and the alkali metal orthophosphate functioning as a precipitant builder, it is somewhat surprising that a mixture of the materials functions so effectively. In particular, when sodium orthophosphate is used alone as a detergency builder it tends to cause inorganic deposition on the washed fabrics, which is accompanied by soil redeposition; but in the presence of the sodium tripolyphosphate the soil redeposition is significantly decreased whilst the detergency is apparently boosted. Moreover, in comparison with the use of sodium tripolyphosphate alone as detergency builder, the same amount of the mixed sodium tripolyphosphate and alkali metal orthophosphate detergency builders shows improved detergency and soil redeposition properties, especially in hard water.

There have in the past been many suggestions for using mixed phosphate detergency builders, for example mixtures of sodium pyrophosphate and sodium tripolyphosphate, in liquid compositions, particularly those adapted for hard surface cleaning. Moreover, it is well known that the condensed phosphates such as sodium tripolyphosphate tend to degrade in aqueous alkaline conditions at high temperatures, which are often met during

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detergent processing conditions, so as to form other phosphates; for example sodium tri-
polyphosphate tends to break down to give
a mixture of sodium pyrophosphate and
sodium orthophosphate.

However, to the Applicants' knowledge it
has not been proposed hitherto to use the
specific mixtures of sodium tripolyphosphate
and alkali metal orthophosphate in the pro-
portions as set out below in homogeneous
fabric washing liquid detergent compositions
according to the present invention. The forma-
tion of the clear liquid detergent compositions
with much higher detergency builder levels
than has hitherto been possible is particularly
advantageous. Apart from the consumer bene-
fits of lower viscosity and better storage
properties than heterogeneous, suspended
liquid compositions, they can generally be
made more easily.

The ratio of the sodium tripolyphosphate
to the alkali metal orthophosphate in the
detergent composition is from 10:1 to 1:10,
particularly 5:1 to 1:2 parts by weight, pre-
ferably from 4:1 to 2:3, and especially from
3:1 to 1:1 parts by weight. These ratios of
sodium tripolyphosphate to the alkali metal
orthophosphates are chosen to give optimum
detergency building and other washing pro-
perties in relation to the maximum amount
of these ingredients which can be tolerated
in the liquid compositions without loss of
stability.

It will be appreciated that the actual
amounts of sodium tripolyphosphate and
alkali metal orthophosphate are chosen
according to the overall phosphate detergency
builder level which is desired or permitted
in the liquid detergent compositions. How-
ever, it is normal to use a total amount of
sodium tripolyphosphate plus alkali metal
orthophosphate within the range of from 6%
to 35% by weight of the composition, pre-
ferably from about 10% to 25%, and espe-
cially 15% to 25% by weight. It is also pre-
ferred that the individual amount of each of
the sodium tripolyphosphate and the alkali
metal orthophosphate should be at least 4%
and 2% respectively, the latter preferably at
least 4% by weight of the composition.

When higher levels of sodium tripolyphos-
phate are used, i.e. above 12% by weight of
the composition, it is advantageous to use as
the sodium tripolyphosphate a type of material
which hydrates rapidly, as this aids its solu-
bility, particularly for use in compositions
containing higher levels of sodium tripoly-
phosphate. Suitable types of sodium tripoly-
phosphate are commercially available with a
high, i.e. greater than 50%, phase I content,
and they may also be partially or fully pre-
hydrated. Processing conditions, including
mixing temperatures, are of course adjusted
according to the type of sodium tripolyphos-
phate employed.

Apart from the mixed phosphate detergency
builders, the liquid detergent compositions of
the invention contain a detergent active com-
pound, which may be an anionic, nonionic,
amphoteric or zwitterionic detergent active
compound or mixture thereof. Many suitable
detergent active compounds are commercially
available and are fully described in the litera-
ture, for example in "Surface Active Agents
and Detergents", Volumes I and II, by
Schwartz, Perry and Berch.

The preferred detergent compounds which
can be used are synthetic anionic compounds.
These are usually water soluble alkali metal
salts of organic sulphates and sulphonates
having alkyl radicals containing from 8 to 22
carbon atoms, the term alkyl being used to
include the alkyl portion of higher acyl
radicals. Examples of suitable synthetic
anionic detergent compounds are sodium and
potassium primary or secondary alkyl sul-
phates, especially those obtained by sulphating
the higher (C_8-C_{18}) alcohols produced by
reducing the glycerides of tallow or coconut
oil; sodium and potassium alkyl ($C_{10}-C_{20}$)
benzene sulphonates, particularly sodium
linear secondary alkyl ($C_{10}-C_{15}$) benzene
sulphonates, sodium alkyl glyceryl ether sul-
phates, especially those ethers of the higher
alcohols derived from tallow or coconut oil
and synthetic alcohols derived from petro-
leum; sodium coconut oil fatty acid mono-
glyceride sulphates and sulphonates; sodium
and potassium salts of sulphuric acid esters
of higher (C_8-C_{18}) fatty alcohol-alkylene
oxide, particularly ethylene oxide, reaction
products; the reaction products of fatty acids
such as coconut fatty acids esterified with
isethionic acid and neutralised with sodium
hydroxide; sodium and potassium salts of
fatty acid amides of methyl taurine; alkane
primary and secondary monosulphonates such
as those derived by reacting alpha-olefins
(C_8-C_{20}) with sodium bisulphite and those
derived by reacting paraffins with SO_2 and
 Cl_2 and then hydrolysing with a base to
produce a random sulphonate; and olefin
sulphonates, which term is used to describe
the material made by reacting olefins, par-
ticularly alpha-olefins, with SO_3 and then
neutralising and hydrolysing the reaction pro-
duct.

Although in general the sodium salts of the
anionic detergent compounds are cheaper, the
potassium salts may be used to advantage,
particularly in compositions with high levels
of other sodium salts such as sodium tripoly-
phosphate and sodium orthophosphate.

Of the anionic detergent compounds, alkali
metal alkyl ($C_{10}-C_{15}$) benzene sulphonates are
particularly preferred, both for ready avail-
ability and cheapness and also for their advan-
tageous solubility properties.

If desired, nonionic detergent active com-
pounds may be used as the sole detergent

compounds, or preferably in admixture with anionic detergent compounds, especially the alkyl benzene sulphonates. Examples include the reaction products of alkylene oxides, usually ethylene oxide, with alkyl (C_8-C_{22}) phenols, generally 5 to 25 EO; i.e. 5 to 25 units of ethylene oxide per molecule; the condensation products of aliphatic (C_8-C_{18}) primary or secondary alcohols with ethylene oxide, generally 2 to 30 EO, e.g. 6-20 EO, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. It is also possible to use such nonionics which have been prepared by first ethoxylating and subsequently propoxylating the hydroxyl-containing organic radical, e.g. C_8-C_{16} alcohols with EO and PO. Other so-called nonionic detergent active compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulfoxides.

Mixtures of detergent active compounds, for example mixed anionic or mixed anionic and nonionic compounds may be used in the detergent compositions, particularly to impart thereto controlled low sudsing properties. Mixtures of amine oxides and ethoxylated anionic compounds can also be beneficial.

Amounts of amphoteric or zwitterionic detergent active compounds can also be used in the liquid detergent compositions of the invention, but this is not normally desired due to their relatively high cost. If any amphoteric or zwitterionic detergent active compounds are used it is generally in small amounts in compositions based on the much more commonly used anionic and/or nonionic detergent active compounds.

The amount of the detergent active compound or compounds used is generally in the range of from 2% to 20%, preferably 5% to 15%, by weight of the compositions, depending on the desired properties. Lower levels of nonionic detergent compounds should be used within this range, as they tend to form a separate liquid phase if used at higher levels, that is over 5% by weight. The ratio of the total detergent active compounds to the total of the amount of sodium tripolyphosphate and alkali metal orthophosphate should generally be in the range of from 2:1 to 1:5, preferably 1:1 to 1:3 parts by weight.

In order to facilitate the production of the homogeneous liquid detergent compositions with relatively high levels of detergency builders present, it is preferred to use potassium rather than sodium orthophosphate. Potassium orthophosphate can be added as such, or orthophosphoric acid may be neutralised by a potassium salt such as potassium hydroxide during production of the compositions. It should be understood that the term alkali metal orthophosphate includes the mono-, di- and tri-orthophosphates.

It is essential to have a hydrotrope system

in the compositions, to reduce any tendency to separation into different liquid phases. The hydrotrope system which has been found effective for use in the homogeneous liquid detergent compositions comprises a hydrotrope, selected from the group consisting of alkali-metal or ammonium toluene-, cumene- and xylene sulphonates, urea, lower aliphatic C_1-C_4 monohydric alcohols such as ethanol which is commonly supplied as industrial methylated spirit (IMS), and a hydrotrope selected from the group consisting of lower (C_8-C_{18}) fatty acid alkylolamides such as coconut ethanolamide, and readily water-soluble soaps of $C_{10}-C_{22}$ fatty acids, including such polymerised fatty acids, such as potassium coconut soap and potassium oleate, potassium soap of dimerized oleic acid and mixtures of these materials. The presence of such soaps as hydrotropes is particularly beneficial in homogeneous liquid compositions containing higher levels of sodium tripolyphosphates, i.e. over 15% by weight. The fatty acid alkylolamides also function as lather boosters and therefore should not normally be used in low sudsing compositions.

The amount of each of the hydrotropes, constituting the hydrotrope system, is normally in the range from 1% to 15%, preferably 5% to 10% by weight of the composition. It should be noted that an excess of these hydrotropes can be deleterious just as well as too little material, for optimum liquid composition properties, but satisfactory levels can be found readily within the range described.

It is desirable to include one or more anti-deposition agents in the liquid detergent compositions of the invention, to decrease any tendency to form inorganic deposits on washed fabrics, especially under conditions of use as low product concentration. Examples of preferred anti-deposition agents are homo- and copolyacrylates, e.g. sodium polyacrylate, the sodium salt of copolymethacrylamide/acrylic acid and sodium poly-alpha-hydroxyacrylate, salts of copolymers of maleic anhydride with ethylene, methyl vinyl ether or styrene, especially 1:1 copolymers, optionally with partial esterification of the carboxyl groups, and the sodium salts of polymaleic acid and polyitaconic acid. Such copolymers preferably have relatively low molecular weights, e.g. in the range of 2,000 to 50,000. The maleic anhydride copolymers can also have some stabilising properties especially when nonionic detergent compounds are used. Other, less preferred, anti-deposition agents include phosphate esters of ethoxylated aliphatic alcohols, polyethylene glycol phosphate esters, and certain phosphonates such as sodium ethane-1-hydroxy-1,1-diphosphonate, ethylenediamine tetramethylene phosphonate and sodium 2-phosphonobutane tricarboxylate. The most preferred anti-deposition agent is sodium polyacrylate having a MW of 2,000

to 30,000, e.g. 15 to 25,000. The amount of such anti-deposition agents is generally 0.1% to 5.0%, preferably 0.2% to 2% by weight of the compositions.

- 5 It is also possible to include in the detergent compositions of the invention minor amounts, for example up to 10% by weight, of other detergency builders, which may be either so-called precipitant builders or
- 10 sequestrant builders. This may be of particular benefit where it is desired to increase detergency whilst using particularly low levels of the essential sodium tripolyphosphate and alkali metal orthophosphate builders, so as to
- 15 achieve particularly low phosphorus contents in the detergent compositions for environmental reasons. Examples of such other detergency builders are amine carboxylates, such as sodium nitrilotriacetate. However, it is
- 20 desirable to have no other phosphate detergency builders present other than the sodium tripolyphosphate and alkali metal orthophosphate, and in particular it is desirable to exclude pyrophosphates from the compositions
- 25 as they tend to increase inorganic deposition and soil redeposition. Some pyrophosphate may be present for example as an impurity in the sodium tripolyphosphate, or it may be produced by hydrolysis of the sodium tripolyphosphate during detergent processing particularly under alkaline conditions at elevated
- 30 temperatures, so low levels of sodium pyrophosphate may be unavoidable, but it is preferred to have no more than 5%, especially less than 2% by weight of pyrophosphate
- 35 present in the compositions.

- The liquid detergent compositions of the invention can contain any of the conventional additives in the amounts in which such additives are normally employed in liquid fabric washing detergent compositions. Examples of these additives include lather depressants, anti-redeposition agents such as carboxymethylcellulose (CMC), alkaline salts such
- 40 as alkali metal carbonate or alkali metal hydroxides, and, usually present in very minor amounts, fluorescent agents, perfumes, enzymes such as proteases and amylases, germicides and colourants. When CMC is
- 45 used it has been found advantageous to incorporate also a stabilising agent for this material, e.g. a hydrogenated castor oil (castor wax), carnauba wax, beeswax, etc.
- 50 The balance of the compositions is water, which is usually present to the extent of 40%, to 75% by weight, preferably 45% to 65% by weight.

- 55 To ensure effective detergency the liquid detergent compositions should be alkaline,

and it is preferred that they should provide a pH within the range of 9 to 12, preferably pH 9.5 to 11 when used in aqueous solution of the composition at the recommended concentration. To meet this requirement, the undiluted liquid composition should also be of high pH, for example pH 12 to 13, preferably about pH 12.5. It should be noted that an excessively high pH, e.g. over pH 13 is less desirable for domestic safety. The ingredients in any such highly alkaline detergent composition should of course be chosen for alkaline stability, especially for pH-sensitive materials such as enzymes, and a particularly suitable proteolytic enzyme in this respect is available under the trade name "Esperase"®.

60 It is desirable to include in the compositions an alkaline buffer, for example alkali metal carbonate, to maintain the pH of at least 9 during use, particularly under conditions of use, for example in hard water or at low product concentrations, when the alkali metal orthophosphate is precipitated in the form of its calcium salt and cannot itself then act as an alkaline buffer. Alkali metal silicates, for example sodium ortho-, meta- or preferably neutral or alkaline silicates, which are commonly used as alkaline buffers are more difficult to incorporate in the homogeneous liquid compositions, especially at higher sodium tripolyphosphate levels. An alkali metal hydroxide, may alternatively or additionally be used to provide an initially high pH.

The liquid detergent compositions can be made easily by adding the ingredients to water in conventional detergent processing equipment. It is preferred for higher levels of phosphate builders to heat the water first, e.g. to 60°C to 80°C, and then to add the inorganic detergency builders, followed by the detergent active compound or compounds and then other inorganic materials. Alternatively, if desired, the heat of reaction between acidic and alkaline ingredients may be used to raise the temperature and facilitate mixing of the ingredients.

65 The resultant compositions have viscosities in the range from 20 to 2,000, especially 50 to 1,000 centipoise, as measured at room temperature in a Brookfield viscometer (Spindle No. 3, 30 rpm).

70 The invention is illustrated by the following Examples in which parts and percentages are by weight except where otherwise indicated.

Examples I to X.

75 A series of stable homogeneous liquid detergent compositions were made to the following formulations:

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Percentages

Ingredient	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6	Ex 7	Ex 8	Ex 9	Ex 10
Sodium alkyl benzene sulphonate ¹	6	6	6	6	6	6	7.5	7.5	6	6
Tallow fatty amide — 11 EO	2	2	2	2	2	-	-	-	2	2
Sodium tripolyphosphate	10	10	10	10	10	10	10	10	13	15
Potassium orthophosphate	10	10	10	10	10	10	7.5	7.5	6	7.5
Coconut fatty acid ²	-	-	2	5	5	5	11	11	-	-
Potassium hydroxide	2	2	3	4	2	2	4	4	1	1
Dimerised oleic acid ³	2	2	2	2	-	-	-	-	5	5
Sodium xylene sulphonate	5	5	5	5	5	5	3	5	5	5
Ethanol	-	2	2	2	2	5	2.0	-	-	-
Water	63	61	58	54	58	60	55	55	62	58.5

¹ In Examples 1 to 8 the alkyl benzene sulphonate had a (C₁₁-C₁₅) alkyl group and in Examples 9 and 10 an alkyl (C₁₁-C₁₂) benzene sulphonate was used.

² Neutralised by some of the KOH to form potassium coconut soap in the composition.

³ Obtained as EMPOL, used for lather control, neutralised by the KOH in the composition.

All these formulations had good detergency properties due to the relatively high detergency building properties of the sodium triphosphate and potassium orthophosphate.

Example XI.

A clear, homogeneous low-sudsing liquid detergent composition was made to the following formulation:

	Ingredient	%	Ingredient	%	
	Sodium alkyl (C_{11} — C_{12}) benzene sulphonate	6.0	Potassium alkyl benzene sulphonate ¹	8.0	
	Tallow fatty amide - 11 EO	2.0	Alkanol (C_9 — C_{11}) - 8 EO	2.0	45
5	Dimerised oleic acid	5.0	Potassium hydroxide	4.5	
	Sodium tripolyphosphate	10.0	Sodium tripolyphosphate	16.5	
	Potassium orthophosphate	6.0	Potassium orthophosphate ²	5.0	
	Sodium xylene sulphonate	5.0	Coconut fatty diethanolamide	2.0	
	Potassium hydroxide	1.0	Potassium coconut soap ¹	1.0	50
10	Potassium carbonate	5.0	Sodium xylene sulphonate/toluene sulphonate (4:1)	6.0	
	Water	60.0	Water	55.9	

This liquid composition was readily pourable and was stable on storage down to about -10°C . Replacement of the potassium carbonate by sodium carbonate also gave a clear stable liquid but of higher viscosity. In both cases the alkaline buffering effect of the carbonate improved the detergent properties of the composition.

20 Example XII.

A homogeneous clear built liquid composition was prepared to the following formulation:

	Ingredient	%
25	Potassium alkyl benzene sulphonate	7.50
	Alkyl (C_{16} — C_{18}) - 25 EO	1.45
	Alkyl (C_{13} — C_{15}) - 11 EO	0.80
	Dimerised oleic acid	5.50
	Sodium tripolyphosphate	13.00
30	Potassium orthophosphate ¹	7.70
	Sodium xylene sulphonate	6.10
	Fluorescent agent	0.15
	Water	57.80

35 ¹ Made by reaction between orthophosphoric acid and potassium hydroxide during production of the composition.

This composition had a low viscosity and was found to be stable down to -4°C .

Example XIII.

40 A clear isotropic high-sudsing built liquid detergent composition was prepared to the following formulation:

¹ Calculated in acid form.

² Formed from dipotassium hydrogen orthophosphate by reaction with the potassium hydroxide.

This product was found to have good stability (-5°C to 52°C) and a low viscosity of about 100 to 200 centipoise at room temperature (Brookfield Spindle No. 3, 30 rpm) at pH 12.5. Replacement of the nonionic detergent compound with other similar materials, e.g. alkanol (C_9 — C_{11}) - 6 EO, alkanol (C_{12} — C_{15}) 7 — EO or alkanol (C_{12} — C_{15}) - 3 EO was found to be equally satisfactory.

Example XIV.

A homogeneous clear built liquid composition was prepared to the following formulation:

	Ingredient	%	
	Potassium alkylbenzene sulphonate (C_{11} — C_{12}) (as acid)	6	
	C_{13} — C_{15} linear primary alcohol, condensed with 7 moles of ethylene/propylene oxide (mole ratio 88:12)	2.5	75
	Oleic acid	5.0	
	Sodium tripolyphosphate	16.5	80
	Potassium orthophosphate	6.0	
	KOH	4.0	
	Sodium toluene sulphonate	6.0	
	CMC (sodium salt)	0.25	
	Castor wax	0.25	85
	Fluorescer, dyes, perfume, etc.	0.3	
	Water	53.2	

The viscosity was 300 cp, the pH 12.5, and the product was stable over prolonged standing periods at -5 and $+37^{\circ}\text{C}$.

WHAT WE CLAIM IS:—

- 5 1. A homogeneous, aqueous built liquid detergent composition comprising:
 - a) from 2—20% by weight of a synthetic detergent active compound;
 - 10 b) at least 4% by weight of sodium or tripolyphosphate;
 - c) at least 2% by weight of sodium or potassium orthophosphate, the sum of b) and c) being from 6—35% by weight;
 - 15 d) 1—15% by weight of a hydrotrope selected from alkali metal or ammonium toluene-, xylene-, cumene-sulphonate, urea, lower aliphatic C_1 — C_4 monohydric alcohols and mixtures thereof; and
 - 20 e) 1—15% by weight of a hydrotrope selected from a C_8 — C_{18} fatty acid alkylolamide, a potassium soap of C_{10} — C_{22} fatty acids, and a potassium soap of dimerized C_{10} — C_{22} fatty acids.
- 25 2. A composition according to claim 1, comprising 5—15% by weight of a synthetic detergent active compound.
- 30 3. A composition according to claim 1 or 2, wherein the sum of b) and c) is 15—25% by weight.
4. A composition according to claims 1—3, wherein the weight ratio of b):c) is from 10:1 to 1:10.
5. A composition according to claim 4, 35 wherein the weight ratio of b):c) is from 4:1 to 2:3.
6. A composition according to claim 5, wherein the weight ratio of b):c) is from 3:1 to 1:1.
- 40 7. A composition according to claims 1—6, comprising the hydrotrope d) in an amount of 5—10% by weight.
8. A composition according to claims 1—7, comprising potassium orthophosphate.
- 45 9. A composition according to claims 1—8, comprising at least 12% by weight of sodium tripolyphosphate.
10. A composition according to claim 9, wherein the sodium tripolyphosphate has a phase I content of more than 50% by weight.
- 50 11. A composition according to claims 1—10, comprising a potassium soap of coconut fatty acids or oleic acid.
12. A composition according to claims 1—55 10, comprising a potassium soap of dimerized oleic acid.
13. A composition according to claims 1—12, further comprising 0.1—5% by weight of an anti-deposition agent.
- 60 14. A composition according to claims 1—13, having a pH of 12—13.
15. A composition according to claims 1—14, substantially as described in the Examples.

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